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Article in *Metallurgical and Materials Transactions A* · December 2010

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Thermal Stability of α Phase of Titanium by Using X-Ray Diffraction

MAHMOUD JAFARI, MEHDI VAEZZADEH, and SOGAND NOROOZIZADEH

At room temperature and ambient pressure, crystalline titanium has a hexagonal-close-packed (hcp) lattice and at high temperature appears as a body-centered-cubic (bcc) structure. In fact, the phase transitions of titanium have been investigated under various pressures and temperatures. However, the phase transitions of titanium have been mostly reported at high pressure, while less attention has been paid to various ranges of high temperature. Therefore, in this study, we have considered the thermal stability of α phase of titanium by using X-ray diffraction (XRD) at high temperature. The observed experimental results of the diffraction show that the stability range of α phase varies between room temperature to around 923 K (650 °C).

DOI: 10.1007/s11661-010-0393-1

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I. INTRODUCTION

AT ambient conditions, Ti crystallizes in the hexagonal-close-packed (hcp) structure, called the α phase, having 2 atoms per unit cell and c/a ratio of ~ 1.58 with space group $P6_3/mmc$,^[1,2] and the unit cell parameters are $a = 2.957 \text{ \AA}$ and $c = 4.685 \text{ \AA}$.^[3-5] At ambient pressure and high temperature (1155 K (882 °C)), before reaching the melting temperature, it transforms to the body-centered-cubic (bcc) structure or the β phase having 1 atom per unit cell with space group $Im\bar{3}m$,^[2] and the unit cell parameter is $a = 3.33 \text{ \AA}$.^[6,7] At ambient temperature and high pressure, it undergoes to the ω phase.^[8,9] It should be mentioned that this phase is often called hexagonal phase. Also, it has 3 atoms per unit cell with the space group $P6/mmm$ and c/a ratio of 0.61.^[1,2] Moreover, its unit cell parameters are $a = 4.598 \text{ \AA}$ and $c = 2.822 \text{ \AA}$.^[3,4] However, δ and γ phases are other phases of Ti, which can be described as an orthorhombic distorted bcc structure^[10] and an orthorhombic distorted hcp structure,^[11] respectively.

Titanium is an element with notable features. For example, it is tough, resistant against corrosion, and biocompatible to iron. In addition, it is also used in medicine such as in the construction of artificial joints and bones. Because of its high price, the application of Ti is only limited to special cases, which require excellent performance, and the ratio of toughness to high weight is significant. For example, the turbine engines and different structures of airplanes can be enumerated.

The aim of the present study is identifying the range of the thermal stability of α phase at ambient pressure in air atmosphere. The stable phases that have been considered for Ti are α and β . Of course, the intermediate phases (metastable phases) such as ω , δ , and γ have

also been reported. As the phase transition of Ti is based on the effect of changes in pressure, temperature, or both of them, observing the phase diagram^[9,12-14] of the titanium (Figure 1) makes it clear that pure α -Ti at room temperature (and lower than that) is more stable than other phases of Ti (that are ω , δ , γ , and β phases). In fact, from an experimental point of view, it is only the α phase of Ti at room temperature that has been reported more in the literature.^[4-6,15] By increasing the pressure and temperature, we would have the phase transitions of $\alpha \rightarrow \omega$ and also $\alpha \rightarrow \beta$.^[3,9,12,16] Of course, the stability of these phases can be changed in terms of temperature by combining some elements with Ti and obtaining an alloy. For example, Al is a phase stabilizer for α phase, which increases the transformation temperature of $\alpha \rightarrow \beta$, whereas V is a stabilizer of β phase, which lowers the transformation temperature.^[17]

According to the schematic phase diagram^[9,12] in Figure 1, the temperature transformation of $\alpha \rightarrow \beta$ is 1155 K (882 °C), which is compatible with our experimental results. Moreover, in this diagram, changes in pressure and temperature would lead to transition phase in a way that, with an increase in pressure or temperature, we would have the transformation of $\alpha \rightarrow \omega$ or $\alpha \rightarrow \beta$. In the equilibrium line (which is represented in the diagram as a solid line) with an increase in pressure, the phase transformation of $\alpha \rightarrow \omega$ can be observed. On the way back, releasing the pressure (with a simple extrapolation) would theoretically convert the ω phase into α at the temperature of 60 K (-213 °C),^[9] which is lower than can be expected to have ω phase. However, it was experimentally proved that with a decrease in pressure, the ω phase does exist in the equilibrium line (solid line in Figure 1); *i.e.*, it has not been totally returned to the α phase at the equilibrium line but at a higher one (the dot-dashed line in Figure 1). In other words, hysteresis will be created (that is, the ω phase will still be retained in titanium) and, in fact, the ω phase also exists at a temperature less than 380 K (107 °C). Having heated to this temperature, the aforementioned phase will disappear. It should be further pointed out

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Manuscript submitted May 5, 2009.

Article published online September 8, 2010

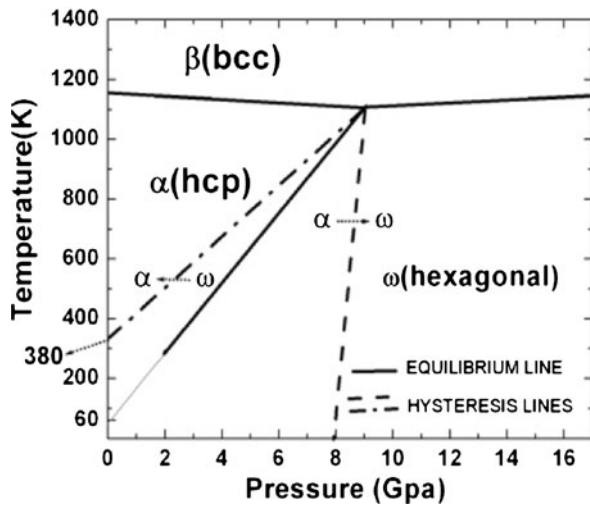


Fig. 1—Schematic phase diagram of titanium.

that some other specific data^[9] of phase diagram can also be added as follows:

$$P_t = 9 \text{ GPa and } T_t = 1100 \text{ K, } T_{\alpha \leftrightarrow \beta} = 1155 \text{ K,}$$

$$T_{\omega \rightarrow \alpha} = 380 \text{ K, and } P_{0 \alpha \leftrightarrow \omega} = 2 \text{ GPa}$$

where P_t and T_t represent the pressure and temperature of the triple point of the phase diagram, respectively. Moreover, $T_{\alpha \leftrightarrow \beta}$ and $T_{\omega \rightarrow \alpha}$ represent, correspondingly, the temperatures at which the α phase transforms into β and also ω phase transforms into α . In addition, $P_{0 \alpha \leftrightarrow \omega}$ represents the pressure at which the transformation of the α to ω takes place.

Based on Figure 1, it can be interpreted that the domain of the titanium phases would change in the forms observable in Figure 1 between the dot-dashed line and dashed line, and we have a mixture of ω and α phases between these two lines. In fact, the existence of this mixture of two phases on this diagram is in agreement with the X-ray diffraction (XRD) pattern obtained (Figure 2).

II. EXPERIMENTAL METHOD

The PHILIPS* PW3710 model of the X-ray diffrac-

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tometer was used under vacuum in order to obtain the Ti XRD data, varying in a range between the room temperature to the highest possible temperature. Titanium powders were taken with a purity of 99.99 pct. The titanium was subjected to *in situ* at a high-temperature X-ray diffraction (HTXRD) experiment to observe the stability of the sample as a function of temperature and also to investigate the eventual formation of metastable phases (such as δ and γ) and other phases of Ti such as ω and β .

The HTXRD patterns were obtained in the 2θ range 14 to 72 deg. The step $\Delta 2\theta$ was 0.02, and the total

measuring time was 0.3 s/step. Moreover, having Ni as a filter, the radiation used was Cu K_α with a mean wavelength of $\lambda = 1.54118 \text{ \AA}$ at 40 kV high voltage and 30 mA of the X-ray tube. The kind of monochromator used was curve graphite and the X-ray detector was proportional, with xenon gas. In addition, the θ - 2θ scan mode used was continuous. Furthermore, the temperature was controlled *via* a heat controller and the accuracy of the temperature measurements was $\pm 1 \text{ }^\circ\text{C}$.

The XRD patterns were scanned in the temperature range between 298 K and 1183 K (25 $^\circ\text{C}$ and 910 $^\circ\text{C}$). The sample was held for 10 minutes at a given temperature and heated to the next temperature, by a heating rate of 293 K (20 $^\circ\text{C}$)/min. Once the temperature of 298 K (25 $^\circ\text{C}$) was reached, a diffraction pattern of the α phase was collected in the 2θ angular range 14 to 72 deg. In order to detect the Ti-phase transformations, the sample temperature was then increased to 753 K, 923 K, 1003 K, 1051 K, and 1183 K (480 $^\circ\text{C}$, 650 $^\circ\text{C}$, 730 $^\circ\text{C}$, 778 $^\circ\text{C}$, and 910 $^\circ\text{C}$).

III. RESULTS AND DISCUSSION

As mentioned earlier, to obtain the Ti XRD data, Ti powders with a purity of 99.99 pct were used and the experiment was carried out at the temperature range varying between the room temperature and the highest temperature that we could possibly achieve in this study. The XRD patterns recorded for all experimental temperatures are shown in Figure 2. Making a comparison between the patterns at the temperature of 298 K (25 $^\circ\text{C}$) with the structure of α phase^[18] from the Joint Committee on Powder Diffraction Standards (JCPDS),^[19] we have observed most of the XRD reflections of the α phase at this temperature. The main reflections of this phase are at the angles of $2\theta = 35.066 \text{ deg}$, 38.405 deg , 40.153 deg , 53.012 deg , 62.965 deg , and 70.663 deg , which are related to the crystal planes of $(10\bar{1}0)$, (0002) , $(10\bar{1}1)$, $(10\bar{1}2)$, $(11\bar{2}0)$, and $(10\bar{1}3)$, and the interplane distance of $d_{hkl} = 2.557 \text{ \AA}$, 2.342 \AA , 2.244 \AA , 1.726 \AA , 1.475 \AA , and 1.332 \AA , respectively. However, at $2\theta = 47 \text{ deg}$, a very slight and insignificant reflection was observed for which we could not make any comments due to the fact that, even making a comparison with the JCPDS Powder Diffraction File,^[19] we could notice that, for the same radiation Cu K_α , it contained no observable reflection for $2\theta = 47 \text{ deg}$ at this specific position.

Figure 2 also shows that, until the temperature has reached 923 K (650 $^\circ\text{C}$), the Ti structure still consists of α phase and there are no phase transitions in the crystal structure. In fact, a decrease in the intensity of the reflections shows that Ti is in the process of phase transition along with an increase in the temperature. In other words, the decrease in XRD reflection intensity may be the result of an increase in the thermal motion of the Ti atoms with an increase in the temperature. However, it seems that a decrease in the reflection intensities and the formation of a new reflection can be considered as proof for the formation of a new transition phase, because the new reflections that are

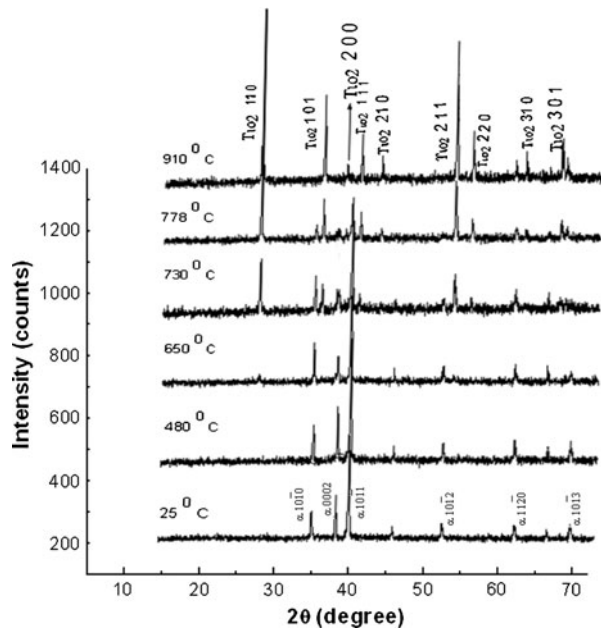


Fig. 2—HTXRD in all range temperatures for Ti.

formed are actually located in new 2θ positions that are different from those that are already known for the α phase.

Analyzing and comparing these results with those of Daniel Errandonea and Sven P. Rudin^[15,20] reveals that the existence of a reflection in the angle of $2\theta = 67.16$ deg in the aforementioned experimental XRD pattern (Figure 2) can be related to the possibility of the formation of ω phase in this range and exactly related to atomic plane (300)/(211) of this phase. Moreover, the combination and formation of several phases in this range have been predicted by the theory of reconstructive phase transitions^[21,22] and also in the phase diagram schematic mentioned earlier. In addition, it has been observed that the stability of the α phase is maintained between room temperature up to 923 K (650 °C), because with an increase in temperature, the aforementioned phase would gradually disappear and lead to the formation of titanium oxide (TiO_2) with the mineralogical name of rutile and the unit cell parameters $a = 4.6001 \text{ \AA}$ and $c = 2.9654 \text{ \AA}$,^[23] which is clearly observable at the temperature of 1003 K (730 °C) and is completed at 1183 K (910 °C). This result is, in fact, in good agreement with the theoretical predictions of References 9, 12, and 21. In the temperature of 1183 K (910 °C), the reflections of TiO_2 are well represented and the reflections are indexed with the JCPDS Powder Diffraction File.^[19]

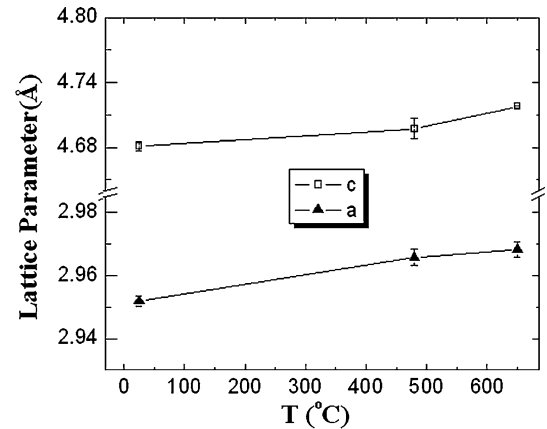
Moreover, by using d and hkl for most of the main reflections of α phase, and also by using the following Eq. [1] for hcp structure,^[24] we could calculate the unit cell parameters, as shown in Table I.

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad [1]$$

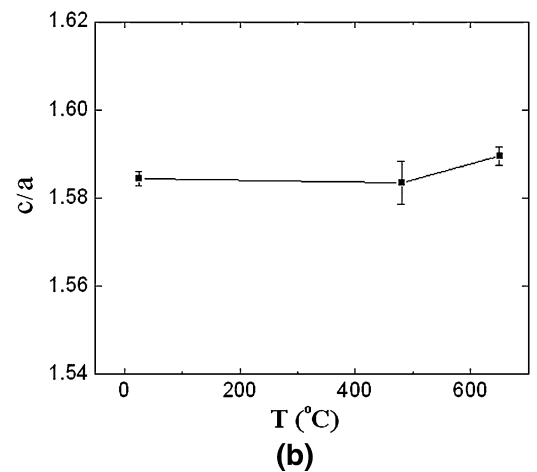
In addition, as shown in Figure 3(a), the lattice parameters, a , on the one hand, and c , on the other, are in a

Table I. Data of Unit Cell Parameters for α -Ti; Estimated Standard Deviations (ESDs) are Indicated

T (K (°C))	$a \pm \text{ESD}$ (Å)	$c \pm \text{ESD}$ (Å)	$c/a \pm \text{ESD}$
298 (25)	2.952 ± 0.002	4.681 ± 0.004	1.585 ± 0.002
753 (480)	2.966 ± 0.003	4.697 ± 0.010	1.584 ± 0.005
923 (650)	2.968 ± 0.002	4.718 ± 0.001	1.590 ± 0.002



(a)



(b)

Fig. 3—(a) Unit cell parameters vs temperature for α -Ti (lines are shown as a guide for eyes). (b) Ratio of c/a parameters vs temperature for α -Ti (lines are shown as a guide for eyes).

linear dependence on temperature. Therefore, c/a also somehow shows a slight increase in terms of temperature from 1.585 at ambient temperature to 1.590 at 923 K (650 °C) (Figure 3(b)). This finding is actually in good agreement with both the theoretical^[25] and the experimental reports.^[3-5,15]

IV. CONCLUSIONS

The experimental results of the HTXRD patterns show that the stability range of α phase varies between room temperature to around 923 K (650 °C). However, with an increase in temperature, this phase would gradually disappear and lead to the formation of

titanium Oxide (TiO₂), which is clearly observable at the temperature of 1003 K (730 °C) and is completed at 1183 K (910 °C).

Furthermore, a non-negligible reflection in the angle of $2\theta = 67.16$ deg was observed, which may be due to the possibility of the formation of ω phase in this position of 2θ , as predicted by the theory.

Finally, the lattice parameters of the α phase shows an increase in terms of temperature in the range investigated.

ACKNOWLEDGMENTS

We express our sincere gratitude to Professor Kamran Ahmadi, Director of the XRD Laboratory of Materials and Energy Research Center in Karaj (Iran), for kindly providing us with several opportunities to benefit from the XRD facilities at the Center.

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